

PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF ELECTION
(PCT Rule 61.2)

Date of mailing (day/month/year)
26 February 2001 (26.02.01)

To:

Commissioner
US Department of Commerce
United States Patent and Trademark
Office, PCT
2011 South Clark Place Room
CP2/5C24
Arlington, VA 22202
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

International application No.	Applicant's or agent's file reference
PCT/FI00/00581	50147
International filing date (day/month/year)	Priority date (day/month/year)
28 June 2000 (28.06.00)	29 June 1999 (29.06.99)

Applicant
MARTIKAINEN, Mika

1. The designated Office is hereby notified of its election made:

in the demand filed with the International Preliminary Examining Authority on:

24 January 2001 (24.01.01)

in a notice effecting later election filed with the International Bureau on:

2. The election was
 was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland	Authorized officer R. E. Stoffel
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI 00/00581

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: C01G 51/04

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: C01G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5569444 A (PHILIPPE BLANCHARD ET AL), 29 October 1996 (29.10.96), column 3, line 47 - line 61, claim 1, abstract --	1-13
A	US 5057299 A (JEAN A. LOWN), 15 October 1991 (15.10.91), claim 1, abstract -- -----	1-13

Further documents are listed in the continuation of Box C.

See patent family annex.

- * Special categories of cited documents
- "A" document defining the general state of the art which is not considered to be of particular relevance
- "B" earlier document but published on or after the international filing date
- "C" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "D" document referring to an oral disclosure, use, exhibition or other means
- "E" document published prior to the international filing date but later than the priority date claimed
- "F" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "G" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "H" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- "I" document member of the same patent family

Date of the actual completion of the international search

24 October 2000

Date of mailing of the international search report

26-10-2000

Authorized officer

Moa Grönkvist/ELY

Telephone No. +46 8 782 25 00

Name and mailing address of the ISA/
Swedish Patent Office
Box 5055, S-102 42 STOCKHOLM
Facsimile No. +46 8 666 02 86
Form PCT/ISA/210 (second sheet) (July 1992)

PATENTTI- JA REKISTERIÄLLITUS
Patentti- ja innovaatiolinja

TUTKIMUSRAPORTTI

PATENTTIHAKEMUS NRO	LUOKITUS
991478	C 01 G 51/04

TUTKITTU AINEISTO

Patenttijulkaisukokonaisuus (FI, SE, NO, DK, DE, CH, EP, WO, GB, US), tutkitut luokat
C 01 G

Tiedonhaut ja muu aineisto

epoqué:wpi,epodoc,paj

VIITEJULKAISUT

Kategoria*)	Julkaisun tunnistetiedot	Koskee vaatimuksia
X	US-A 5569444 (C 01 G 51/00)	1-13

*) X Patentoitavuuden kannalta merkittävä julkaisu yksinään tarkasteltuna
Y Patentoitavuuden kannalta merkittävä julkaisu, kun otetaan huomioon tämä ja yksi tai useampi samaan kategoriaan kuuluva julkaisu
Δ Yleistä tekniikan tasoa edustava julkaisu, ei kuitenkaan patentoitavuuden este

Päiväys 11.1.2000	Tutkija M.Koskela
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PATENT COOPERATION TREATY

PCT

REC'D 08 OCT 2001

WIPO PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 50147/IR	FOR FURTHER ACTION		See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No. PCT/FI00/00581	International filing date (day/month/year) 28.06.2000	Priority date (day/month/year) 29.06.1999	
International Patent Classification (IPC) or national classification and IPC7 C 01 G 51/04			
Applicant OMG Kokkola Chemicals Oy et al			

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 4 sheets, including this cover sheet.

This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 1 sheets.

3. This report contains indications relating to the following items:

- I Basis of the report
- II Priority
- III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV Lack of unity of invention
- V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI Certain documents cited
- VII Certain defects in the international application
- VIII Certain observations on the international application

Date of submission of the demand 24.01.2001	Date of completion of this report 17.09.2001	
Name and mailing address of the IPEA/SE Patent- och registreringsverket Box 5055 S-102 42 STOCKHOLM Facsimile No. 08-667 72 88	Telex 17978 PATOREG-S	Authorized officer Jan Carlerud/ELY Telephone No. 08-782 25 00

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/FI00/00581

I. Basis of the report

1. With regard to the elements of the international application:*

 the international application as originally filed the description:pages 1-6 , as originally filed

pages _____, filed with the demand

pages _____, filed with the letter of _____

 the claims:

pages _____, as originally filed

pages _____, as amended (together with any statement) under article 19

, filed with the demand

pages _____, filed with the letter of 07.08.2001 the drawings:pages 1 , as originally filed

pages _____, filed with the demand

pages _____, filed with the letter of _____

 the sequence listing part of the description:

pages _____, as originally filed

pages _____, filed with the demand

pages _____, filed with the letter of _____

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language English which is: the language of a translation furnished for the purposes of international search (under Rule 23.1(b)). the language of publication of the international application (under Rule 48.3(b)). the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

 contained in the international application in written form. filed together with the international application in computer readable form. furnished subsequently to this Authority in written form. furnished subsequently to this Authority in computer readable form. The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished. The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.4. The amendments have resulted in the cancellation of: the description, pages _____ the claims, Nos. _____ the drawings, sheet/fig. _____5. This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2 (c)).**

* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).

** Any replacement sheet containing such amendments must be referred to under item I and annexed to this report.

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/FI00/00581

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims	1-9	YES
	Claims	_____	NO
Inventive step (IS)	Claims	1-9	YES
	Claims	_____	NO
Industrial applicability (IA)	Claims	1-9	YES
	Claims	_____	NO

2. Citations and explanations (Rule 70.7)

Documents cited in the International Search Report:

A) US 5569444 A
 B) US 5057299 A

This Report refers to the amended claims filed 7 August 2001.

The present invention relates to a hydroxide of cobalt or of cobalt and some other metal. It is intended to provide a product with high density and a large particle size.

Document A describes a hydroxide of cobalt, nickel and cadmium or zinc and its production. The main metal is nickel and the proportion of cobalt in the hydroxide is 1-8%. Ammonium ions are added to a solution of nitrates or sulphates of said metals in an amount such that the ratio of complexing agent to metal is approximately within the interval 0,5-3 specified in claim 1. The temperature is maintained between 80 and 85 degrees centigrade and the pH is regulated to a value of 9,2 +/- 0,1.

The present claim 1 does not specify the proportions of the metals in the hydroxide. However, it specifies the product as "cobaltous hydroxide or... ...of cobalt and some other metal". Thus, the invention is different from what is disclosed in A in that the main metal in the hydroxide is cobalt. Furthermore, the pH is 10-13 and the mixture is not heated.

Document B, which is cited in the description, is cited as a further example of prior art technique.

Therefore, the claimed invention is novel.

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/FI00/00581

Supplemental Box

(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: V.

In view of what is disclosed in the cited documents, the cobaltous hydroxide cannot be considered to be obvious to a person skilled in the art. Therefore, the invention is considered to involve an inventive step. It is also considered to be industrially applicable.

Claims

1. Cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal, **characterised** in having a density of about 0.5-2.2 g/cm³, a particle size above about 1 µm, typically about 1-20 µm, and a specific surface of about 0.5-20 m²/g, and that it is prepared by adding a complexing agent and hydroxide ion under alkaline conditions to an aqueous chloride solution of cobalt or to an aqueous chloride solution of an alloy of cobalt and some other metal in order to form metal hydroxide, wherein the complexing agent is selected so as to form an ammonium complex with the metal ion, the molar ratio of complexing agent to metal being approx. 0.5-3 and the pH being adjusted in the range 10-13.
2. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 1, **characterised** in that the pH is regulated in the range 11.2-12.0.
3. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 1, **characterised** in that the other metal is nickel, manganese, magnesium or aluminium, or alloys of these.
4. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 1, **characterised** in that the concentration of the chloride solution is in the range from 10 to 120 g/l calculated on the total metal content.
5. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 1, **characterised** in that the complexing agent is ammonium sulphate or aqueous ammonia
6. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 1, **characterised** in that the molar ratio of complexing agent to metal is approx. 1.5-2.
7. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 1, **characterised** in that NaOH is used for pH regulation.
8. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 1, **characterised** in that the reaction is carried out at a temperature of about 40-90 °C.
9. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 8, **characterised** in that the reaction is carried out at a temperature of about 70 °C.

PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

Berggren Oy Ab
P.O. Box 16
FIN-00101 Helsinki
Finland

Berggren Oy Ab

19-06-2001

PCT

WRITTEN OPINION

(PCT Rule 66)

Date of mailing
(day/month/year)

15-06-2001

Applicant's or agent's file reference
50147/IR

REPLY DUE

within 60 days
from the above date of mailing

International application No.
PCT/FI00/00581

International filing date (day/month/year)
28.06.2000

Priority date (day/month/year)
29.06.1999

International Patent Classification (IPC) or both national classification and IPC7
C 01 G 51/04

Applicant
OMG Kokkola Chemicals Oy et al

14.8.01

1. This written opinion is the first (first, etc.) drawn by this International Preliminary Examining Authority.

2. This opinion contains indications relating to the following items:

- I Basis of the report
- II Priority
- III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV Lack of unity of invention
- V Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI Certain documents cited
- VII Certain defects in the international application
- VIII Certain observations on the international application

3. The applicant is hereby invited to reply to this opinion.

When? See the time limit indicated above. The applicant may, before the expiration of that time limit, request this Authority to grant an extension, see Rule 66.2(d).

How? By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3. For the form and the language of the amendments, see Rules 66.8 and 66.9.

Also For an additional opportunity to submit amendments, see Rule 66.4. For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4bis. For an informal communication with the examiner, see Rule 66.6.

If no reply is filed, the international preliminary examination report will be established on the basis of this opinion.

4. The final date by which the international preliminary examination report must be established according to Rule 69.2 is: 29.10.2001

Name and mailing address of the IPEA/SE

Patent- och registreringsverket
Box 5055
S-102 42 STOCKHOLM

Facsimile No. 08-667 72 88

Telex
17978
PATOREG-S

Authorized officer

Jan Carlerud/ELY
Telephone No. 08-782 25 00

I. Basis of the opinion

1. With regard to the elements of the international application:*

 the international application as originally filed the description:

pages _____, as originally filed

pages _____, filed with the demand

pages _____, filed with the letter of _____

 the claims:

pages _____, as originally filed

pages _____, as amended (together with any statement) under article 19

pages _____, filed with the demand

pages _____, filed with the letter of _____

 the drawings:

pages _____, as originally filed

pages _____, filed with the demand

pages _____, filed with the letter of _____

 the sequence listing part of the description:

pages _____, as originally filed

pages _____, filed with the demand

pages _____, filed with the letter of _____

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item. These elements were available or furnished to this Authority in the following language English which is: the language of a translation furnished for the purposes of international search (under Rule 23.1(b)). the language of publication of the international application (under Rule 48.3(b)). the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the written opinion was drawn on the basis of the sequence listing:

 contained in the international application in printed form. filed together with the international application in computer readable form. furnished subsequently to this Authority in written form. furnished subsequently to this Authority in computer readable form. The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished. The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.4. The amendments have resulted in the cancellation of: the description, pages _____ the claims, Nos. _____ the drawings, sheet/fig _____5. This opinion has been drawn as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2 (c)).

* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed".

V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims	1-10	YES
	Claims	11-13	NO
Inventive step (IS)	Claims		YES
	Claims	1-13	NO
Industrial applicability (IA)	Claims	1-13	YES
	Claims		NO

2. Citations and explanations

Documents cited in the International Search Report:

- A) US 5569444 A
- B) US 5057299 A

The present invention relates to a method for preparing a hydroxide of cobalt or of cobalt and some other metal with high density and a large particle size and to the product produced with the method.

Document A describes a method for producing hydroxide of cobalt, nickel and cadmium or zinc. Ammonium ions are added to a solution of nitrates or sulphates of said metals in an amount such that the ratio of complexing agent to metal is approximately within the interval 0,5-3 specified in claim 1. The pH is regulated to a value of 9,2 +/- 0,1.

Thus, the claimed method is different from what is disclosed in A in that the pH is 10-13. It is, however, considered to be an obvious measure to a person skilled in the art to perform experiments to find out what effect changes to parameters such as pH, temperature or concentrations have to the product. Therefore, the method of claims 1-10 is not considered to involve an inventive step.

The product specified in claims 11-13 does not differ from the product disclosed in A, see column 3, lines 47-61. Therefore, the invention of claims 11-13 lacks novelty.

Document B, which is cited in the description, is cited as a further example of prior art technique.

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:

Claim 13 refers to claims 11 and 12, which both are independent claims. This is not allowed. A dependent claim can refer only to one independent claim.

7 August 2001

Toimitusjohtaja
Managing Director
L. Nordin

• PATENTIT

• SOYLLISYYSMALLIT

• PATENTS

UTILITY MODELS

J. Kupiainen
M. Brax
E. Heikkinen
T. Läkki
B. Lassénus
T. Pelin
I. Risku
O-P. Saiponmaa
J. Svensson
P. Täthua
B. Träskman
J. Joronen
M. Karttunen
S. Kuusma
M. Laajalahti
V. Tognetti
S. Ylätalo

• MALLIT

• DESIGNS

N. Mikander
L. Valjakka

• TAVARAMERKIT
LAKIASIAT
• TRADEMARKS

LEGAL MATTERS

P. Kolve**
H. Halmetoja**
S. Henn**
I. Karlsson**
E-M. Söderström**
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FAX: +46-8-667 7288 (5 pages)
Confirmation by mail

Authorized Officer: Jan Carlerud/ELY
Our ref: 50147/IR/MG

REPLY TO WRITTEN OPINION
INTERNATIONAL PATENT APPLICATION PCT/FI00/00581
APPLICANT:OMG KOKKOLA CHEMICALS OY

On account of the Written Opinion issued on 15 June 2001 we submit the following:

We enclose a new claim set directed to the cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal, i.e. to the product itself. The aqueous brine has been amended to a chloride solution as illustrated in the examples.

In applications of the accumulator industry the capacity of the accumulators can be raised by using cobaltous hydroxide with maximum density as an additive in accumulators. In addition it is important that the cobaltous hydroxide used has a small specific surface and a high density. The cobaltous hydroxide of claim 1 has both and also large particle size.

The conventional cobaltous hydroxide is very fine-grained and because of this small particle size it also has large spesific surface.

The larger particle size, and specifically the possibility for adjusting the particle size, is achieved by choice of the complexing agent and process parameters, as in claim 1.

In the method of US patent 5,057,299 the mixture is heated and kept in hydrothermal conditions until the cobaltous hydroxide precipitates. With the method of this US patent, the particle size increases at higher temperatures. The method of the patent allows the particle size to be controlled only in the range from approx. 0.05 to 0.5 μm by means of the temperature. Temperature is not critical parameter in the present invention.

Berggren Oy Ab

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FINLAND

• European Patent Attorney
• European Trademark Attorney

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Kotipaikka Helsinki

Berggren

In the US patent 5,569,444 a method for preparing nickel hydroxide is presented. There may be only 8 % cobalt among the nickel. Further solutions of nitrates or sulphates are used in the US patent, whereas in the present invention chloride solution is used. Also the pH is lower in the US patent than in the present invention.

The cobaltous hydroxide of claim 1 has considerably larger particle size than the metal hydroxides of the cited prior art, and this is achieved by choice of the complexing agent and process parameters, as in claim 1.

BERGGREN OY AB



Ira Risku
Patent Agent

Encls

a set of new claims 1 to 9 in triplicate

Claims

1. Cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal, characterised in having a density of about 0.5-2.2 g/cm³, a particle size above about 1 µm, typically about 1-20 µm, and a specific surface of about 0.5-20 m²/g, and that it is prepared by adding a complexing agent and hydroxide ion under alkaline conditions to an aqueous chloride solution of cobalt or to an aqueous chloride solution of an alloy of cobalt and some other metal in order to form metal hydroxide, wherein the complexing agent is selected so as to form an ammonium complex with the metal ion, the molar ratio of complexing agent to metal being approx. 0.5-3 and the pH being adjusted in the range 10-13.
2. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 1, characterised in that the pH is regulated in the range 11.2-12.0.
3. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 1, characterised in that the other metal is nickel, manganese, magnesium or aluminium, or alloys of these.
4. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 1, characterised in that the concentration of the chloride solution is in the range from 10 to 120 g/l calculated on the total metal content.
5. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 1, characterised in that the complexing agent is ammonium sulphate or aqueous ammonia
6. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 1, characterised in that the molar ratio of complexing agent to metal is approx. 1.5-2.
7. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 1, characterised in that NaOH is used for pH regulation.
8. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 1, characterised in that the reaction is carried out at a temperature of about 40-90 °C.
9. A cobaltous hydroxide or alloy hydroxide formed of cobalt and some other metal as defined in claim 8, characterised in that the reaction is carried out at a temperature of about 70 °C.

RECORD COPY

1/4

50147

PCT REQUESTOriginal (for **SUBMISSION**) - printed on 28.06.2000 09:26:26 AM

0-1	For receiving Office use only International Application No.	PCT/FI 00 / 00581
0-2	International Filing Date	28 JUN 2000 (28-06-2000)
0-3	Name of receiving Office and "PCT International Application"	The Finnish Patent Office PCT International Application
0-4 0-4-1	Form - PCT/RO/101 PCT Request Prepared using	PCT-EASY Version 2.90 (updated 10.05.2000)
0-5	Petition The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty	
0-6	Receiving Office (specified by the applicant)	National Board of Patents and Registration (Finland) (RO/FI)
0-7	Applicant's or agent's file reference	50147
I	Title of invention	PROCESS FOR MAKING HIGH DENSITY AND LARGE PARTICLE SIZE COBALT HYDROXIDE OR COBALT MIXED HYDROXIDES AND A PRODUCT MADE BY THIS PROCESS
II	Applicant This person is:	applicant only
II-1	Applicant for	all designated States except US
II-4	Name	OMG KOKKOLA CHEMICALS OY
II-5	Address:	P.O. Box 286 FIN-67101 Kokkola Finland
II-6	State of nationality	FI
II-7	State of residence	FI
III-1	Applicant and/or inventor This person is:	applicant and inventor
III-1-1	Applicant for	US only
III-1-4	Name (LAST, First)	MARTIKAINEN, Mika
III-1-5	Address:	Hakalahdenkatu 83 B 22 FIN-67100 Kokkola Finland
III-1-6	State of nationality	FI
III-1-7	State of residence	FI

PCT REQUEST

Original (for SUBMISSION) - printed on 28.06.2000 09:26:26 AM

IV-1	Agent or common representative; or address for correspondence The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: Name Address:	agent BERGGREN OY AB P.O. Box 16 FIN-00101 Helsinki Finland +358-9-693701 +358-9-6933944 email.box@berggren.fi
V	Designation of States	
V-1	Regional Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AP: GH GM KE LS MW MZ SD SL SZ TZ UG ZW and any other State which is a Contracting State of the Harare Protocol and of the PCT EA: AM AZ BY KG KZ MD RU TJ TM and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT EP: AT BE CH&LI CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE and any other State which is a Contracting State of the European Patent Convention and of the PCT OA: BF BJ CF CG CI CM GA GN GW ML MR NE SN TD TG and any other State which is a member State of OAPI and a Contracting State of the PCT
V-2	National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH&LI CN CR CU CZ DE DK DM DZ EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT TZ UA UG US UZ VN YU ZA ZW
V-5	Precautionary Designation Statement In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation(s) of the State(s) indicated under item V-6 below. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit.	
V-6	Exclusion(s) from precautionary designations	NONE

PCT REQUEST

Original (for SUBMISSION) - printed on 28.06.2000 09:26:26 AM

VI-1	Priority claim of earlier national application		
VI-1-1	Filing date	29 June 1999 (29.06.1999)	
VI-1-2	Number	991478	
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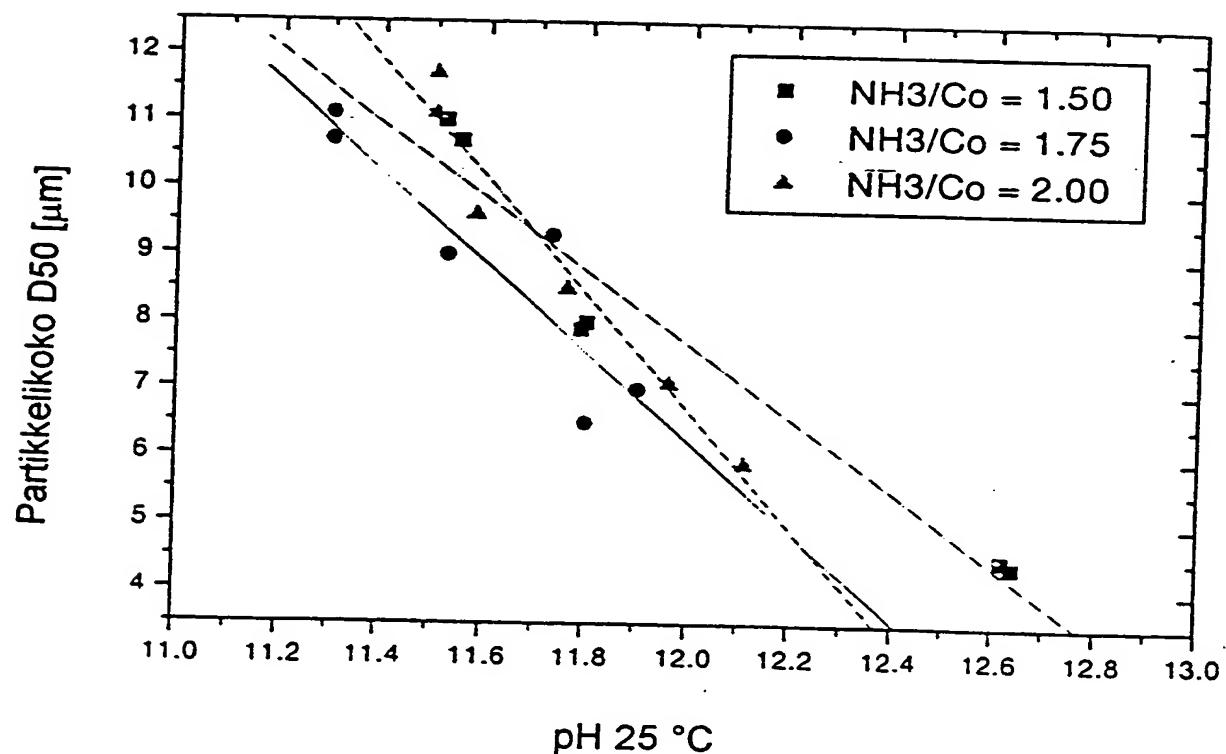
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Kuva 1

Menetelmä korkean tilavuuspainon ja suuren partikkelikoon omaavan koboltti-hydroksidin tai kobolttimetalliseoshydroksidin valmistamiseksi ja menetelmällä saatu tuote

5 Keksintö koskee menetelmää korkean tilavuuspainon ja suuren partikkelikoon omaavan kobolttihydroksidin tai koboltin ja jonkin muun metallin seoshydroksidin valmistamiseksi. Keksintö koskee myös tällä menetelmällä aikaansaatua tuotetta.

Kobolttihydroksidia käytetään monissa sovellutuksissa, esimerkiksi elektroniikka-teollisuudessa lisääineena ladattavissa NiMH- ja NiCd-akuissa. Lisäksi se soveltuu käytettäväksi oksidituotteiden, kuten LiCoO_2 ja LiCoMO_2 , valmistuksessa prekursorina (M tarkoittaa metallia). Kobolttihydroksidia käytetään myös katalyyttinä tai prekursorina katalyyttituotannossa. Edellä mainittuihin tarkoituksiin soveltuu käytettäväksi myöskin hydroksidituote, jossa koboltin ohella on jotain muuta metallia, kuten nikkeliä, mangaania, mangnesiumia tai alumiinia.

15 Alalla tunnetaan monia menetelmiä kobolttihydroksidin valmistamiseksi. US-patentin 5 057 299 mukaisessa menetelmässä kobolttihydroksidia valmistetaan yhdistämällä koboltti-ioni kompleksoivan aineen kanssa, jolloin muodostuu vesiliukoinen kobolttikompleksi. Tähän lisätään hydroksidi-ioni ja seosta kuumennetaan ja pideätään hydrotermaalisissa olosuhteissa, kunnes kobolttihydroksidi saostuu. Tämän US-patentin menetelmällä partikkelikoko saadaan kasvamaan korkeampia lämpötiloja käytettäessä. Partikkelikokoa voidaan patentin mukaisella menetelmällä lämpötilan avulla säätää kuitenkin vain välillä noin 0,05-0,5 μm .

Akkuteollisuuden sovellutuksissa NiCd- ja NiMH-akuita vaaditaan hyvää kapasiteettia. Kapasiteettia voidaan nostaa käytämällä akkujen lisääineena kobolttihydroksidia, jonka tilavuuspaino on mahdollisimman korkea. Lisäksi oksidituotteiden valmistuksessa on tärkeää, että käytetyllä kobolttihydroksilla on alhainen ominaispinta-ala ja korkea tilavuuspaino. Esillä olevan keksinnön tarkoituksesta on saada aikaan menetelmä, jolla voidaan valmistaa suuren partikkelikoon omaavia koboltti-hydroksidi-partikkeleita tai koboltin ja jonkin muun metallin seoshydroksidipartikkeleita helposti kontrolloitavalla menetelmällä. Tarkoituksesta on saada aikaan kobolttihydroksidipartikkeleita tai koboltin ja jonkin muun metallin seoshydroksidi-partikkeleita, joiden partikkelikoko on yli 1 μm , edullisesti yli 3 μm . Tämä on keksinnön mukaisesti saatu aikaan siten kuin on esitetty oheisissa patenttivaatimuksissa.

Keksinnön mukaisella menetelmällä voidaan valmistaa paitsi kobolttihydroksidia myös koboltin ja jonkin muun metallin tai metalleiden seoshydroksidia. Muina metalleina voidaan käyttää esimerkiksi nikkeliä, mangaania, mangnesiumia tai aluminia tai näiden seoksia käyttötarkoitukseen mukaan.

5 Keksinnön mukaisessa menetelmässä käytetään lähtöaineena koboltin tai koboltin ja seosmetallien vesipitoisia suolaliuoksia sulfaatin, nitraatin tai kloridin muodossa. Liuoksen konsentraatio voi vaihdella välillä 10-120 g/l kokonaismetallipitoisuuden suhteen laskettuna.

10 Tähän metallisuolaliuokseen lisätään sellaista kompleksinmuodostaja-ainetta, joka muodostaa ammoniumkompleksin metalli-ionin kanssa. Kompleksinmuodostaja-aine voi olla ammoniumsulfaattia, vesipitoista ammoniakkia tai jokin muu ammoniumilähde. Periaatteessa kompleksinmuodostaja-aine voi olla myös jotain muuta metallin kanssa kompleksin muodostavaa ainetta, kuten esimerkiksi EDTA:ta. Esillä olevassa eksinnössä ammoniumioni on kuitenkin havaittu hyväksi kompleksinmuodostaja-aineeksi sen halpuuden ja hyvien kompleksinmuodostusominaisuksiensa takia. Reaktoriin syötettävän ammoniumkompleksinmuodostaja-aineen ja metallin välinen moolisuhde on edullisesti noin 0,5-3, edullisimmin noin 1,5-2,0. Reaktio suoritetaan alkalisissa olosuhteissa ja pH:n säätämiseen käytetään edullisesti NaOH:ia. Edullinen pH-arvo on välillä 10-13, edullisimmillaan pH on välillä 11,2-12,0. Reaktio suoritetaan lämpötilassa noin 40-90 °C, edullisesti lämpötilassa noin 70 °C. Keksinnön mukaisella menetelmällä saadaan kobolttihydroksidia, jonka tilavuuspaino on noin 0,5-2,2 g/cm³, partikkelikoko yli noin 1 µm, tyypillisesti noin 1-20 µm ja ominaispinta-ala noin 0,5-20 m²/g.

15 20 Keksinnön mukaisella menetelmällä saadut partikkelit ovat muodoltaan heksagonaalisia levymäisiä kobolttihydroksidipartikkeleita. Keksinnön yhteydessä havaittiin, että kloridiliuoksilla saadaan paksumpia hiukkasia kuin sulfaattipohjaisilla liuoksilla, kun muut reaktio-olosuhteet pidetään samoina. Paksumpien hiukkasten katsotaan soveltuvan paremmin esimerkiksi litiumkobolttioksidin valmistukseen.

25 30 Keksinnön mukaisessa menetelmässä kobolttihydroksidin partikkelikokoa voidaan säätää ammoniumionin ja koboltti-ionin moolisuhteen avulla ja käytetyn pH:n avulla. Kuvassa 1 on esitetty partikkelikoon muutos pH:n funktiona erilaisilla NH₃-ioni/Co-suhteilla. Kuvasta nähdään, että pH:n nostaminen pienentää partikkelikokoa, ja että mitä korkeampi on NH₃-ioni/Co-suhde sitä merkittävämpi vaiketus pH:lla on partikkelikokoon.

Seuraavassa keksintöä on valaistu esimerkkien avulla. Esimerkeissä 1-3 on menetelmä suoritettu vertailuna ilman kompleksinmuodostusta ammoniumionin kanssa. Esimerkeissä 4-6 on kuvattu keksinnön mukainen menetelmä, jossa muodostetaan ammoniumkompleksi koboltin kanssa. Esimerkissä 7 on kuvattu keksinnön mukainen menetelmä, jossa on käytetty koboltin lisäksi nikkelia. Esimerkeissä on reaktiolämpötilana käytetty 70 °C.

Vertailuesimerkki 1

10 CoCl₂:n vesiliuosta (30 g/l Co) syötettiin jatkuvasti ensimmäiseen reaktoriin. pH pidettiin vakioarvossa 11,8-12,0 lisäämällä natriumhydroksidiliuosta. Ensimmäisen reaktorin ylivirtaus johdettiin toiseen reaktoriin, jossa virtaukseen lisättiin natriumhydroksidia siten, että pH oli 13,5. Toisen reaktorin ylivirtaus suodatettiin ja pestiin vedellä. Pesty Co(OH)₂-kakku kuivattiin.

15 Kuivatun Co(OH)₂:n keskimääräinen partikkelikoko D50 oli 0,9 µm mitattuna Malvern Mastersizer -partikkelikokoanalyaattorilla (mittaus suoritettiin laserdiffraktiolla vesipitoisesta Co(OH)₂-lietteestä). Tilavuuspaino oli 0,5 g/cm³ (ASTM B527-93). Ominaispinta-ala (BET ASTM D4567-86) oli 30 m²/g.

Vertailuesimerkki 2

20 CoCl₂:n vesiliuosta (30 g/l Co) syötettiin jatkuvasti ensimmäiseen reaktoriin. pH pidettiin vakioarvossa 11,6-11,8 lisäämällä natriumhydroksidiliuosta. Ensimmäisen reaktorin ylivirtaus johdettiin toiseen reaktoriin, jossa virtaukseen lisättiin natriumhydroksidia siten, että pH oli 13,5. Toisen reaktorin ylivirtaus suodatettiin ja pestiin vedellä. Pesty Co(OH)₂-kakku kuivattiin.

25 Kuivatun Co(OH)₂:n keskimääräinen partikkelikoko D50 oli 1,1 µm mitattuna Malvern Mastersizer -partikkelikokoanalyaattorilla (mittaus suoritettiin laserdiffraktiolla vesipitoisesta Co(OH)₂-lietteestä). Tilavuuspaino oli 0,7 g/cm³ (ASTM B527-93). Ominaispinta-ala (BET ASTM D4567-86) oli 29 m²/g.

Vertailuesimerkki 3

30 CoCl₂:n vesiliuosta (30 g/l Co) syötettiin jatkuvasti ensimmäiseen reaktoriin. pH pidettiin vakioarvossa 11,2-11,4 lisäämällä natriumhydroksidiliuosta. Ensimmäisen reaktorin ylivirtaus johdettiin toiseen reaktoriin, jossa virtaukseen lisättiin natriumhydroksidia siten, että pH oli 13,5. Toisen reaktorin ylivirtaus suodatettiin ja pestiin vedellä. Pesty Co(OH)₂-kakku kuivattiin.

Kuivatun Co(OH)_2 :n keskimääräinen partikkeli koko D50 oli $1,9 \mu\text{m}$ mitattuna Malvern Mastersizer -partikkeli kokoanalysoattorilla (mittaus suoritettiin laserdiffraktiolla vesipitoisesta Co(OH)_2 -lietteestä). Tilavuuspaino oli $0,6 \text{ g/cm}^3$ (ASTM B527-93). Ominaispinta-ala (BET ASTM D4567-86) oli $35 \text{ m}^2/\text{g}$.

5 Esimerkki 4

CoCl₂:n vesiliuosta (30 g/l Co) syötettiin jatkuvasti ensimmäiseen reaktoriin, jossa oli ammoniakin vesiliuosta siten, että metallin moolisuhde ammoniumiin oli 2. pH pidettiin vakioarvossa 11,8-12,0 lisäämällä natriumhydroksidiliuosta. Ensimmäisen reaktorin ylivirtaus johdettiin toiseen reaktoriin, jossa virtaukseen lisättiin natriumhydroksidia siten, että pH oli 13,5. Toisen reaktorin ylivirtaus suodatettiin ja pestiin vedellä. Pesty Co(OH)₂-kakku kuivattiin.

Kuivatun Co(OH)_2 :n keskimääräinen partikkeli koko D50 oli $1,8 \mu\text{m}$ mitattuna Malvern Mastersizer -partikkeli kokoanalysoattorilla (mittaus suoritettiin laserdiffraktiolla vesipitoisesta Co(OH)_2 -lietteestä). Tilavuuspaino oli $0,7 \text{ g/cm}^3$ (ASTM B527-93). Ominaispinta-ala (BET ASTM D4567-86) oli $5,8 \text{ m}^2/\text{g}$.

Esimerkki 5

CoCl₂:n vesiliuosta (30 g/l Co) syötettiin jatkuvasti ensimmäiseen reaktoriin, jossa oli ammoniakin vesiliuosta siten, että metallin moolisuhde ammoniumiin oli 2. pH pidettiin vakioarvossa 11,6-11,8 lisäämällä natriumhydroksidiliuosta. Ensimmäisen reaktorin ylivirtaus johdettiin toiseen reaktoriin, jossa virtaukseen lisättiin natriumhydroksidia siten, että pH oli 13,5. Toisen reaktorin ylivirtaus suodatettiin ja pestiin vedellä. Pesty Co(OH)₂-kakku kuivattiin.

Kuivatun Co(OH)_2 :n keskimääräinen partikkeli koko D50 oli $3,9 \mu\text{m}$ mitattuna Malvern Mastersizer -partikkeli kokoanalysoattorilla (mittaus suoritettiin laserdiffraktiolla vesipitoisesta Co(OH)_2 -lietteestä). Tilavuuspaino oli $1,2 \text{ g/cm}^3$ (ASTM B527-93). Ominaispinta-ala (BET ASTM D4567-86) oli $2,6 \text{ m}^2/\text{g}$.

Esimerkki 6

CoCl₂:n vesiliuosta (30 g/l Co) syötettiin jatkuvasti ensimmäiseen reaktoriin, jossa oli ammoniakin vesiliuosta siten, että metallin moolisuhde ammoniumiin oli 2. pH pidettiin vakioarvossa 11,2-11,4 lisäämällä natriumhydroksidiliuosta. Ensimmäisen reaktorin ylivirtaus johdettiin toiseen reaktoriin, jossa virtaukseen lisättiin natrium-

hydroksidia siten, että pH oli 13,5. Toisen reaktorin ylivirtaus suodatettiin ja pestiin vedellä. Pesty Co(OH)₂-kakku kuivattiin.

Kuivatun Co(OH)₂:n keskimääräinen partikkelikoko D50 oli 7,4 μm mitattuna Malvern Mastersizer -partikkelikokoanalyaattorilla (mittaus suoritettiin laserdiffraktiolla vesipitoisesta Co(OH)₂-lietteestä). Tilavuuspaino oli 1,7 g/cm³ (ASTM B527-93). Ominaispinta-ala (BET ASTM D4567-86) oli 1,8 m²/g.

Esimerkki 7

Valmistettiin seosmetallisuolaliuos sisältäen CoCl₂- ja NiCl₂-liuoksia (30 g/l Co ja 8 g/l Ni). Liuosta syötettiin jatkuvasti ensimmäiseen reaktoriin, jossa oli ammoniakin vesiliuosta siten, että metallin moolisuhde ammoniumiin oli 2. pH pidettiin vakioarvossa 11,2-11,4 lisäämällä natriumhydroksidiliuosta. Ensimmäisen reaktorin ylivirtaus johdettiin toiseen reaktoriin, jossa virtaukseen lisättiin natriumhydroksidia siten, että pH oli 13,5. Toisen reaktorin ylivirtaus suodatettiin ja pestiin vedellä. Pesty kakku kuivattiin.

15 Kemiallinen analyysi osoitti, että seosmetallihydroksidia oli saostunut. Kuivatun Co_{0,8}Ni_{0,2}(OH)₂:n keskimääräinen partikkelikoko D50 oli 6,9 μm mitattuna Malvern Mastersizer -partikkelikokoanalyaattorilla (mittaus suoritettiin laserdiffraktiolla vesipitoisesta lietteestä). Tilavuuspaino oli 1,6 g/cm³ (ASTM B527-93). Ominaispinta-ala (BET ASTM D4567-86) oli 3,2 m²/g.

20 Vertailun helpottamiseksi alla on esitetty taulukon muodossa edellä kuvattujen esimerkkien tuotteiden fysikaaliset ominaisuudet. Taulukossa on lisäksi esitetty tuotteiden kidekoko mitattuna röntgendiffraktometrillä.

Taulukko 1

Esimerkki	Saostus-pH	Kompleksin-muodostus-aine	Keskimää-räinen partikkeli-koko (μm)	Tilavuus-paino (g/cm ³)	Ominais-pinta-ala (m ² /g)	XRD (001) (nm)	XRD (101) (nm)
Vertailu-esimerkki 1	11,8-12,0	ei mitään	0,9	0,5	30	23	27
Vertailu-esimerkki 2	11,6-11,8	ei mitään	1,1	0,7	29	26	30
Vertailu-esimerkki 3	11,2-11,4	ei mitään	1,9	0,6	35	27	32
Esimerkki 4	11,8-12,0	NH ₃ -ioni	1,8	0,7	5,8	50	49
Esimerkki 5	11,6-11,8	NH ₃ -ioni	3,9	1,2	2,6	56	56
Esimerkki 6	11,2-11,4	NH ₃ -ioni	7,4	1,7	1,8	61	64
Esimerkki 7	11,2-11,4	NH ₃ -ioni	6,9	1,6	3,2	59	57

Taulukosta voidaan nähdä, että käytettäessä kompleksinmuodostaja-aineena ammoniumonia, saatiin tuotteelle suurempi partikkelikoko ja tilavuuspaino kuin ilman kompleksinmuodostajaa. Poikkeuksena oli esimerkin 4 tuote, jonka partikkelikoko ja tilavuuspaino vastasivat suunnilleen vertailuesimerkkien tuotteiden partikkelikokoa ja tilavuuspainoa. Tosin tälläkin tuotteella ominaispinta-ala ja kidekoko vastasivat esimerkkien 5-7 tuotteiden arvoja.

Edellä on esitetty eräitä keksinnön sovelluksia. Keksintöä luonnollisesti ei rajoiteta edellä esitettyihin esimerkkeihin, vaan keksinnön mukaista periaatetta voidaan muunnella patenttivaatimusten suoja-alan puitteissa.

Patenttivaatimukset

1. Menetelmä korkean tilavuuspainon ja suuren partikkelikoon omaavan koboltihydroksidin tai koboltin ja jonkin muun metallin seoshydroksidin valmistamiseksi, jossa koboltin vesipitoiseen suolaliuokseen tai koboltin ja jonkin muun metallin seoksen vesipitoiseen suolaliuokseen lisätään alkalisissa olosuhteissa kompleksinmuodostaja-ainetta ja hydroksidi-iona metallihydroksidin muodostamiseksi, tunnettu siitä, että kompleksinmuodostaja-aine valitaan siten, että se muodostaa ammoniumkompleksin metalli-ionin kanssa, kompleksinmuodostaja-aineen ja metallin välinen moolisuhde on noin 0,5-3, ja pH säädetään välille 10-13.
- 5 10 2. Patenttivaatimuksen 1 mukainen menetelmä, tunnettu siitä, että pH säädetään välille 11,2-12,0.
3. Patenttivaatimuksen 1 mukainen menetelmä, tunnettu siitä, että koboltin tai koboltin ja jonkin muun metallin seoksen suolaliuos on sulfaatin, nitraatin tai kloridin muodossa.
- 15 4. Patenttivaatimuksen 1 mukainen menetelmä, tunnettu siitä, että suolaliuoksen konsentraatio on välillä 10 ja 120 g/l kokonaismetallipitoisuuden suhteen laskettuna.
5. Patenttivaatimuksen 1 mukainen menetelmä, tunnettu siitä, että kompleksinmuodostaja-aine on ammoniumsulfaattia tai vesipitoista ammoniakkia.
- 20 6. Patenttivaatimuksen 1 mukainen menetelmä, tunnettu siitä, että kompleksinmuodostaja-aineen ja metallin välinen moolisuhde on noin 1,5-2.
7. Patenttivaatimuksen 1 mukainen menetelmä, tunnettu siitä, että pH:n säättämiseen käytetään NaOH:ia.
8. Patenttivaatimuksen 1 mukainen menetelmä, tunnettu siitä, että reaktio suoritetaan lämpötilassa noin 40-90 °C.
- 25 9. Patenttivaatimuksen 8 mukainen menetelmä, tunnettu siitä, että reaktio suoritetaan lämpötilassa noin 70 °C.
10. Patenttivaatimuksen 1 mukainen menetelmä, tunnettu siitä, että muina metallina käytetään nikkeilia, mangaania, mangnesiumia tai alumiinia tai näiden seoksia.

11. Kobolttihydroksidi tai koboltin ja jonkin muun metallin muodostama seoshydroksidi, **tunnettu** siitä, että sen tilavuuspaino on noin $0,5\text{-}2,2 \text{ g/cm}^3$, partikkeli-koko yli noin $1 \mu\text{m}$, tyypillisesti noin $1\text{-}20 \mu\text{m}$ ja omiaispinta-ala noin $0,5\text{-}20 \text{ m}^2/\text{g}$.
12. Patenttivaatimuksen 11 mukainen kobolttihydroksidi tai koboltin ja jonkin muun metallin muodostama seoshydroksidi, **tunnettu** siitä, että muu metalli on nikeli, mangaani, mangnesium tai alumiini tai näiden seos.
13. Patenttivaatimuksen 11 tai 12 mukainen kobolttihydroksidi tai koboltin ja jonkin muun metallin muodostama seoshydroksidi, **tunnettu** siitä, että se on valmistettu jollain patenttivaatimusten 1-10 mukaisella menetelmällä.

(57) Tiivistelmä

Keksintö koskee menetelmää korkean tilavuuspainon ja suuren partikkeliin omanavat kobolttihydroksidin tai koboltin ja jonkin muun metallin seoshydroksidin valmistamiseksi, jossa koboltin vesipitoiseen suolaliuokseen tai koboltin ja jonkin muun metallin seoksen vesipitoiseen suolaliuokseen lisätään alkalisissa olosuhteissa kompleksinmuodostaja-ainetta ja hydroksidi-iona metallihydroksidin muodostamiseksi. Menetelmässä kompleksinmuodostaja-aine valitaan siten, että se muodostaa ammoniumkompleksin metalli-ionin kanssa. Kompleksinmuodostaja-aineen ja metallin välinen moolisuhde on noin 0,5-3, ja pH säädetään välille 10-13.